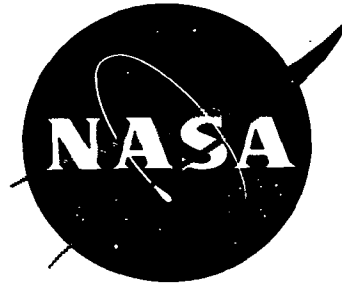


**GULF GENERAL ATOMIC**  
P.O. BOX 608, SAN DIEGO, CALIFORNIA 92112



NASA-CR-120945

Gulf-GA-A12121

**FABRICATION OF (U, Zr) C-FUELED/TUNGSTEN-CLAD SPECIMENS FOR  
IRRADIATION IN THE PLUM BROOK REACTOR FACILITY**

**CASE FILE  
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J. W. R. Creagh**

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Contract: NAS 3-13471**

**Issued: July 12, 1972**

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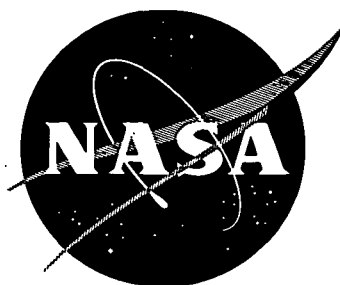
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## PREVIOUS SUMMARY REPORTS

Contract NAS 5-1253	GA-3523, Final Report for the Period Ending August 31, 1962. (U)
Contract NAS 3-2301	GA-3642, Final Report for the Period Ending August 31, 1962. (U)
Contract NAS 3-2532	GA-4769, Final Report for the Period Ending August 31, 1963. Part I (U), Part II (C/RD)
Contract NAS 3-4165	NASA CR-54322, GA-5665, Summary Report for the Period September 1, 1963 through August 31, 1964. (C/RD)
Contract NAS 3-6471	NASA CR-54980, GA-6860, Summary Report for the Period September 1, 1964 through November 23, 1965. (C/RD)
Contract NAS 3-8504	NASA CR-72315, GA-7682, Summary Report for the Period November 23, 1965 through January 31, 1967. (C/RD)
Contract NAS 3-8504	NASA CR-72327, GA-7745, Semi-Annual Report for the Period February 1, 1967 through July 31, 1967. (C/RD)
Contract NAS 3-6471	NASA CR-72517, GA-8974, Summary Report for the Period November 23, 1965 through September 30, 1968. (C/RD)
Contract NAS 3-8504	NASA CR-72627, GA-8956, Summary Report for the Period February 1, 1967 through July 31, 1969. (C/RD)
Contract NAS 3-8504	NASA CR-72947, Gulf-GA-11035, Summary Report for the Period August 1, 1969 through December 7, 1970. (C/RD)
Contract NAS 3-11822	NASA CR-120839, Gulf-GA-A11049, Summary Report for the Period June 1, 1968 through January 31, 1971. (C/RD)

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## INTRODUCTION

Previously under Contract NAS 3-6471, <sup>(1)</sup> two capsules designated as V-2C and V-2D were fabricated for the study of the high temperature irradiation behaviors of carbide fueled tungsten emitter for nuclear thermionic application. V-2C and V-2D were irradiated at NASA Plum Brook Reactor Facility (PBRF) for 11,000 and 12,000 hours respectively at a maximum cladding temperature of 1923°K to burnups of  $3.1 \times 10^{20}$  and  $2.1 \times 10^{20}$  fission/cc. Test results on these capsules have been presented in Reference (2) and (3). Hot cell examinations of these two capsules have been completed and the results will be described in a forthcoming summary report. <sup>(4)</sup>

To continue the evaluation of the irradiation properties of carbide fueled tungsten emitters at high temperatures, two other capsules designated as V-2E and V-2F were assembled under the present contract (NAS 3-13471) for irradiation at PBRF. The stoichiometry and configuration of the carbide fuel bodies and the thickness and operating temperature of the tungsten claddings were selected on the basis of the irradiation data available with the objective of improving the compatibility and dimensional stability of the carbide-tungsten fuel-cladding system under long term irradiation. The



nominal outside diameter and fuel length of the claddings were the same as those described in Reference (1).

This report describes the fabrication and evaluation of the carbide fuel samples and the tungsten claddings used in the fuel pins of these capsules. A list of the previously published reports on thermionic materials work sponsored by NASA at Gulf General Atomic is given on the page next to the title page of this report.

## SUMMARY

90UC-10ZrC fuel samples and chemically vapor deposited tungsten fuel cups were fabricated for the study of the long-term dimensional stability and compatibility of the carbide-tungsten fuel-cladding systems under irradiation. These fuel samples and fuel cups were assembled into the fuel pins of two capsules, designated as V-2E and V-2F, for irradiation in NASA Plum Brook Reactor Facility at a fission power density of 172 watts/c. c. and a maximum cladding temperature of 1823°K. Fabrication methods and characteristics of the fuel samples and fuel cups prepared are described in this report.

## 1. CARBIDE FUEL FABRICATION AND EVALUATION

### 1.1. FUEL COMPOSITION AND STOICHIOMETRY

90UC-10ZrC containing 4 wt% of tungsten was selected as the fuel material for both V-2E and V-2F capsules. This is the same fuel material as that irradiated in V-2C capsule and most of the in-pile converters and thermionic fuel elements tested up-to-date. Tungsten is added to the carbide fuel material to prevent the dissolution of the tungsten cladding by carbide fuel.<sup>(5)</sup> The presence of tungsten additive also improves the stability of the open porosities of the carbide fuel body at high temperatures.<sup>(6)</sup>

The C/U atom ratio of the fuel material prepared was between 1.02 and 1.035, with one carbon atom assigned to each zirconium atom and one carbon atom assigned to every two tungsten atoms. This C/U value was lower than that specified for V-2C capsules (1.03-1.05). It is intended to find out whether the lowering of the C/U atom ratio of the carbide fuel material would improve its compatibility with the tungsten cladding.

## 1.2. CARBIDE FUEL SAMPLE CONFIGURATION AND DIMENSIONS

The configuration and the dimensions of the carbide fuel sample are shown in Fig. 1. The geometric surface area to volume ratio ( $A/V$ ) for this configuration is higher than the highest value used on the fuel samples in the V-2C capsule ( $7.17 \text{ cm}^2/\text{cc.}$  versus  $6.91 \text{ cm}^2/\text{cc.}$ ), where "A" is the geometric surface area of the fuel sample which is available for fission gas release, i. e. the surface area not in contact with the tungsten cladding, the central tungsten thermocouple well and the neighboring carbide samples; while "V" is the volume of the donut-shaped fuel sample minus the volume of the grooves and the holes. These grooves and holes are incorporated into the fuel body in order to facilitate the release of fission gas from the fuel body. The total volume of such grooves and holes amounts to 10% of the volume of the fuel sample before these grooves and holes were machined. It is hoped that the higher ( $A/V$ ) value would help to improve the dimensional stability of the carbide fuel bodies and the tungsten claddings of the fuel pins in V-2E and V-2F capsules.

## 1.3. FUEL ENRICHMENT

The tungsten cladding in the fuel pin of both the V-2E and the V-2F capsules has a thickness of 63 mils, which rejects heat by radiation and conduction across a 28 mil gap filled with neon to an oxidized Inconel surface of high thermal emittance ( $\sim 0.8$ ). Using a gamma heat input of 1 watt/gm

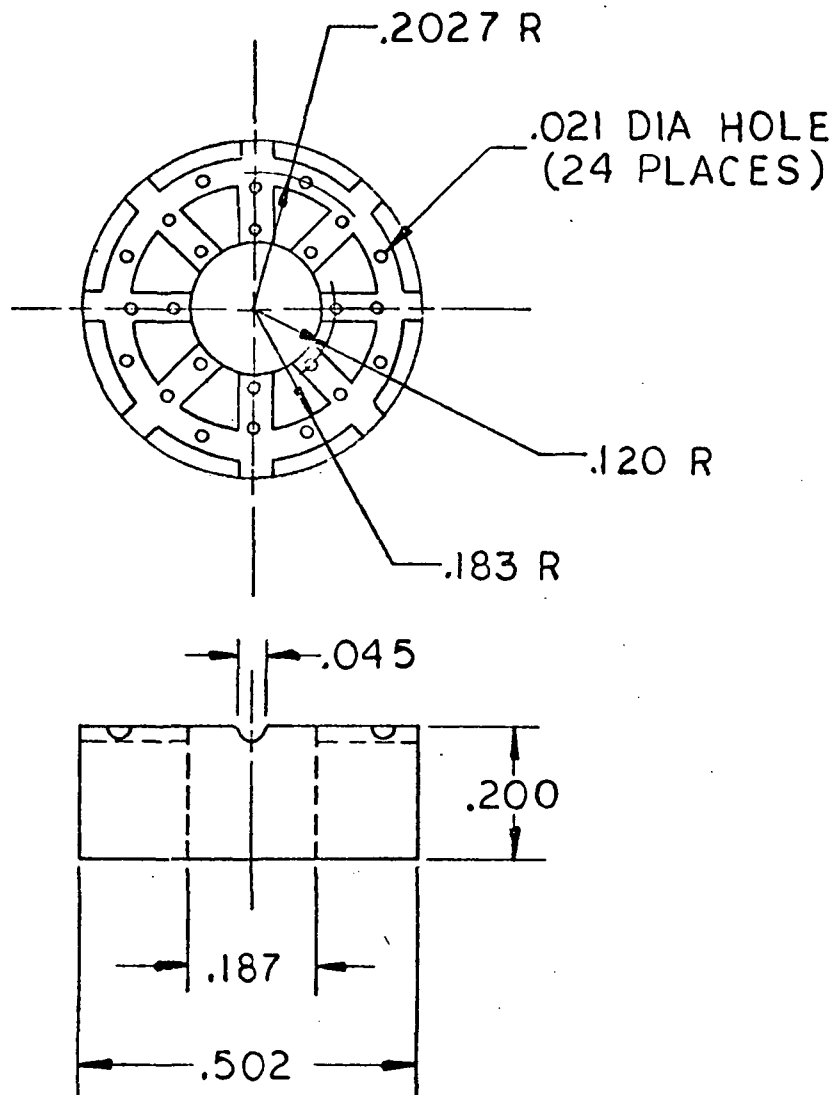


Fig. 1. Configuration and dimensions (in inch) of fuel sample for V-2E and V-2F capsules

and assuming no thermal resistance at the fuel-cladding interface, it was shown that an average fission power density of 172 watts/cc. in the annular space occupied by the fuel sample would be needed in order to maintain the maximum cladding temperature at the designed value of 1823°K. Figure 2 shows the relative fission power density in the fuel samples of V-2E and V-2F capsules as a function of fuel enrichment, with the design fission power density in the fuel sample of V-2C capsule (170 watts/cc.) as the reference.\* Figure 3 shows the radial distribution of the fission power density in the fuel sample for two fuel enrichment values, 30 atom % and 50 atm %. Figure 4 relates the fission power density in the fuel sample to the radial location of the capsule in the V-2 tube. It can be seen from Fig. 2 that an enrichment of 38 atom % is needed to produce the required fission power density (172 watts/cc.) in the fuel sample. However, if the same fuel enrichment (30 atom %) as that in V-2C capsule is used for V-2E and V-2F capsules, the required fission power density could be attained if the capsules were positioned 1/8 inch off the center of the V-2 tube toward the reactor core (see Fig. 4). It was decided that V-2E and V-2F capsules should use the same fuel enrichment (30 atom %) as V-2C capsule since the radial distribution of the fission power density in all these capsules would then be similar.

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\*These calculations were based on a tungsten cladding thickness of 60 mils and assumed to be applicable to the present case. This assumption is justified, since the perturbation is small (60 mils versus 63 mils).

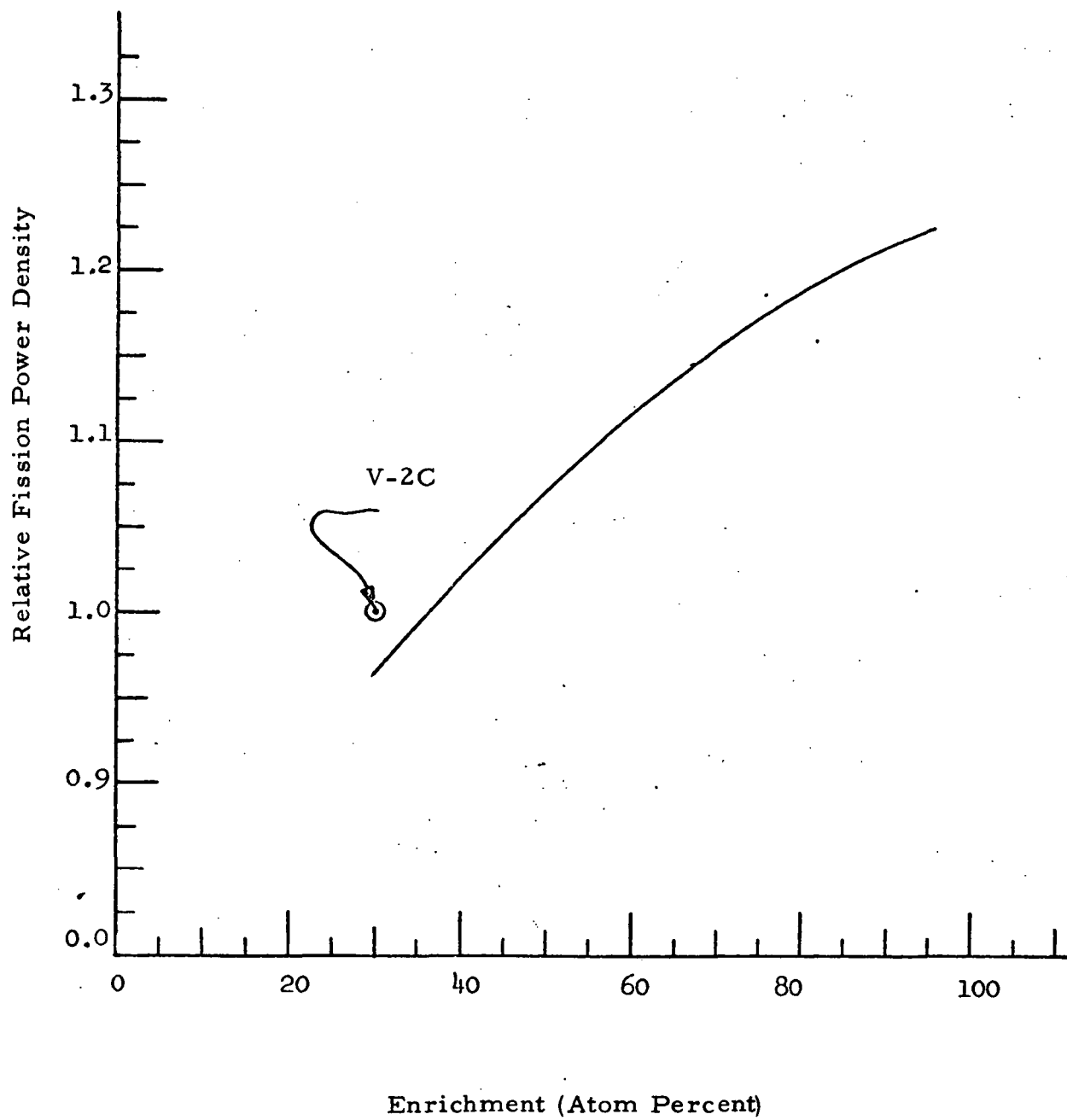


Fig. 2. Relative fission power density as a function of fuel enrichment in V-2E and V-2F capsules

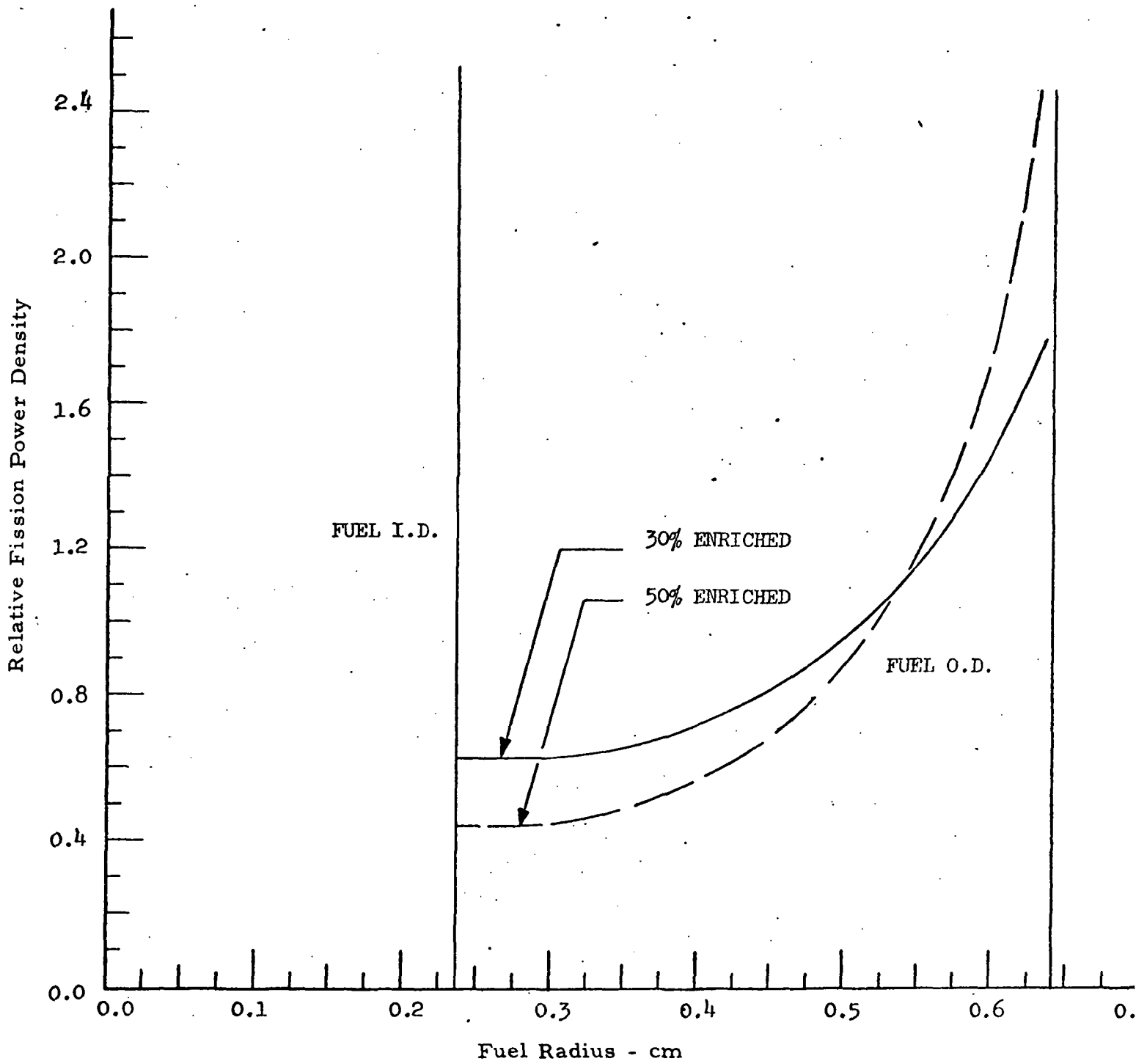


Fig. 3. Radial distribution of relative fission power density in V-2E and V-2F fuel sample



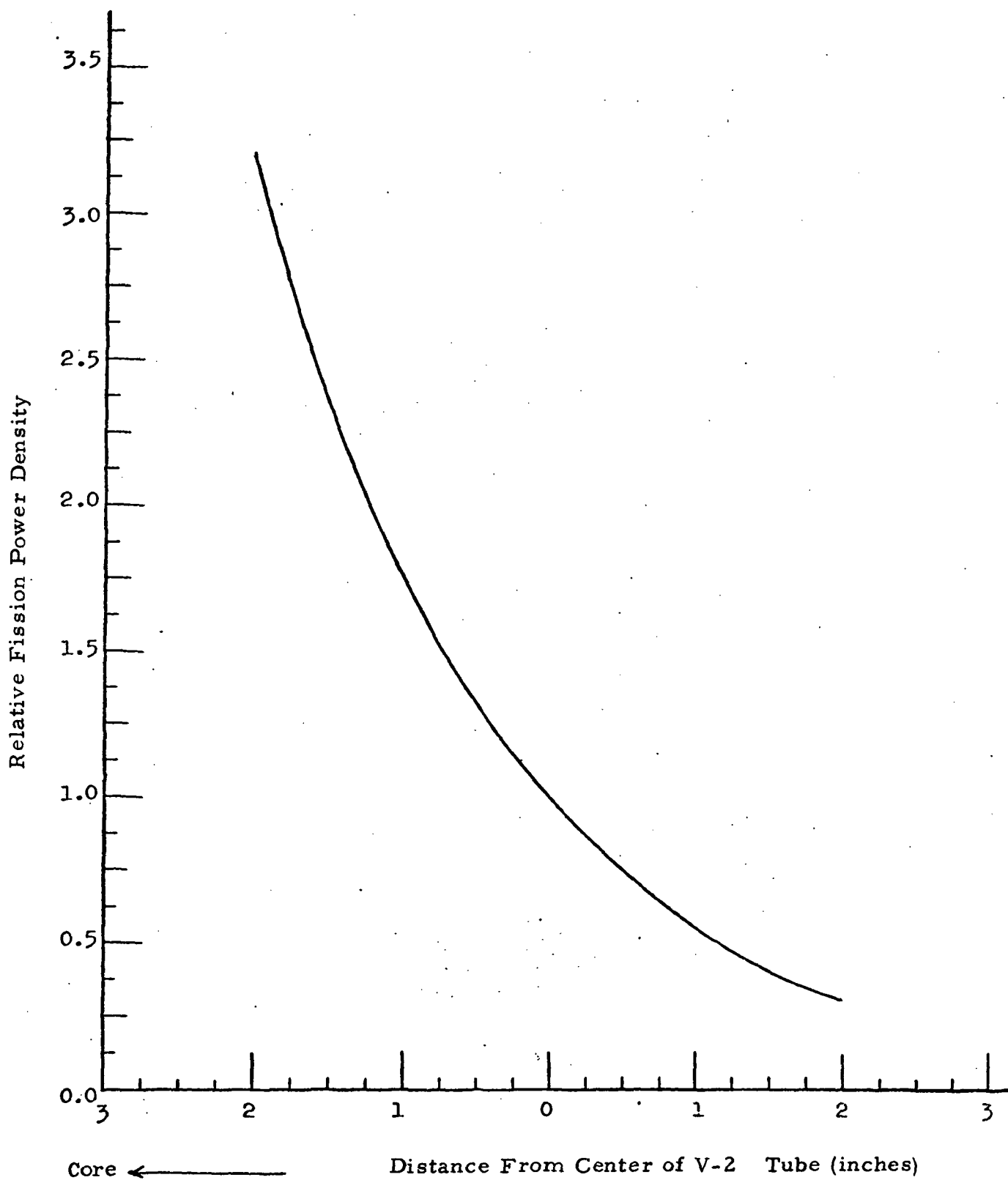


Fig. 4. Relative fission power density in fuel sample of V-2E and V-2F capsules as a function of distance from center of V-2 tube

#### 1.4. FUEL SAMPLE FABRICATION AND EVALUATION

The fuel samples of V-2E and V-2F capsules were fabricated and evaluated according to the following procedures.

- (1) The required amounts of U, Zr and C were arc-melted into carbide buttons in an argon atmosphere.
- (2) The arc-melted carbide buttons were ground and sieved to -44 micron size powder in argon in a glove box, the moisture and oxygen contents of which were each below 10 ppm.
- (3) Tungsten powder of -44 micron size was blended with the carbide powder to achieve a tungsten concentration of 4 weight percent.
- (4) The blended powder was cold-pressed at 80,000 psi in a steel die and the compact was homogenized at  $2558^{\circ}\text{K}$  for 6 hours in a vacuum of  $10^{-5}$  torr or better.
- (5) The composition of the homogenized compact was analyzed to insure that the stoichiometry met the specification ( $\text{C/U} = 1.02$  to  $1.035$ ).
- (6) The acceptable compact was ground and sieved to powder of -44/+20 micron size range in argon in the same glove box as that used in (2).

- (7) The powder from (6) was isostatically cold-pressed at 90,000 psi and the pressed carbide fuel body was sintered at 2343°K for 16 hours in a vacuum of  $10^{-5}$  torr or better.
- (8) The sintered carbide body was centerless ground under oil to the required diameter (see Fig. 1), from which two qualification specimens, each 0.20 inch thick, were obtained.
- (9) These two qualification specimens were outgassed at 2073°K for 50 hours in a vacuum of  $10^{-5}$  torr or better. One of these two specimens was used for the determination of the composition, stoichiometry, and density of the sintered body, and the other was used for the measurement of the true surface area to geometric surface area ratio (SRF value) of the sintered body by the low pressure krypton adsorption technique.
- (10) If the results obtained under (9) met the specification (i. e.  $C/U = 1.02-1.035$ , density = 75-80% of theoretical value, and  $SRF > 350$ ), the sintered carbide body was considered acceptable. The qualified carbide fuel body was outgassed at 2073°K for 50 hours in a vacuum of  $10^{-5}$  torr or better, and machined into fuel pellets according to Fig. 1. Each fuel sample set contained nine fuel pellets, eight of which had grooves on one face only and one of which had grooves on both

faces. When loaded into the fuel cup, the grooved surface of each fuel pellet faced the closed end of the cup, and the one with grooves on both faces occupied the top position of the fuel stack. In doing so, free surface for venting the fission gas was provided between neighboring fuel pellets and between the top fuel pellet and the tungsten radiation shields. In addition to these nine fuel pellets, one additional fuel pellet, designated as "spare" (grooved on one side only), was prepared for the evaluation of the SRF value, composition and stoichiometry of the machined fuel sample set.

- (11) All ten fuel pellets in each fuel sample set were outgassed at 2073°K for 24 hours in a vacuum of  $10^{-5}$  torr or better. The SRF value of the "spare" fuel pellet was then measured. If the SRF value was greater than 160, the degree of open porosity of the fuel sample set was considered as acceptable. One half of the "spare" fuel pellet was then analyzed to insure that the machining operation did not affect the stoichiometry of the fuel material. The other half of the "spare" sample was used for the determination of the enrichment and the oxygen and nitrogen contents of the fuel material.

- (12) Each acceptable fuel sample set was stored in an evacuated glass ampoule prior to loading into the fuel pin of the capsule.

A total of four fuel sample sets was prepared. These were designated as V-2E1, V-2E2, V-2F1 and V-2F2. V-2E1, V-2E2, and V-2F1 were three finished-machined sets, while V-2F2 contained only the central venting hole, with the smaller venting holes remaining to be machined. The evaluation results for these four sets are shown in Table 1. Table 2 contains the results obtained on the metallic impurity contents of V-2E2 and V-2F1 sample sets by emission spectroscopy.

Capsule V-2F was loaded with fuel sample set V-2F1. Capsule V-2E was at first loaded with fuel sample set V-2E1. The fuel pin, however, had to be unloaded because of difficulties encountered during the loading operation. After these difficulties were overcome, V-2E capsule was reloaded with fuel sample set V-2E2. Chemical analysis carried out on one of the nine fuel pellets of fuel sample set V-2E1 after their removal from the fuel pin of V-2E capsule showed that the fuel stoichiometry ( $C/U = 1.02$  and  $1.025$  in duplicate analysis) was still within the specified range of  $C/U = 1.02 - 1.035$ . However, since one of the fuel pellets of this set was consumed by chemical analysis, another fuel pellet was fabricated and evaluated as replacement. The evaluation results for this replacement sample are also shown in Table 1.

TABLE I  
PHYSICAL AND CHEMICAL CHARACTERISTICS OF  
FUEL SAMPLES FOR V-2E AND V-2F CAPSULES

Fuel Sample Set	Qualification Sample				Spare Sample					
	Composition	Stoichiometry (C/U)	Density (% Theoretical)	SRF	Composition	Stoichiometry (C/U)	SRF	Enrichment (wt%)	Oxygen (ppm)	Nitrogen (ppm)
V-2E1	U .4286 Zr .0468 W .0271 C .4978	1.022	78.0	356	U .4300 Zr .0454 W .0260 C .4985	1.023	168	29.67	248	158
V-2E1 Replaced Pellet	U .4286 Zr .0468 W .0271 C .4978	1.022	78.0	356	U .4295 Zr .0457 W .0262 C .4986	1.024	170	29.73	252	168
V-2E2	U .4297 Zr .0454 W .0260 C .4989	1.025	78.2	365	U .4299 Zr .0454 W .0260 C .4987	1.024	165	29.71	243	148
V-2F1	U .4286 Zr .0468 W .0271 C .4978	1.022	78.0	356	U .4273 Zr .0478 W .0266 C .4983	1.023	169	29.15	236	153
V-2F2	U .4297 Zr .0454 W .0260 C .4989	1.025	78.2	365	Sample to be machined					

TABLE 2

SPECTROCHEMICAL ANALYSIS RESULTS FOR  
FUEL SAMPLES IN V-2E AND V-2F CAPSULES

Element	Concentration (ppm)	Element	Concentration (ppm)
Ag	<0.2	Mn	<1.0
Al	<14.0	Mo	<6.0
B	<0.4	Na	<40.0
Be	<1.0	Ni	<6.0
Bi	<1.0	P	<100.0
Ca	<50.0	Pb	<2.0
Cd	<0.3	Rb	<10.0
Co	<30.0	Sb	<100.0
Cr	<8.0	Si	<30.0
Cu	<1.4	Sn	<2.0
Fe	<36.0	Sr	<10.0
In	<4.0	Tl	<0.5
Li	<1.0	V	<10.0
Mg	<4.0	Zn	<100.0

Table 3 lists the dimensions of the fuel pellets of fuel sample sets V-2E2 and V-2F1, which were loaded in V-2E capsule and V-2F capsule respectively.

## 2. TUNGSTEN CLADDING FABRICATION AND EVALUATION

In each fuel pin assembly, the carbide fuel pellets were contained in a tungsten cup of 0.631 inch O. D. and 0.505 inch I. D. The cladding thickness was thus 63 mils and the nominal cold clearance between the I. D. of the cladding and the O. D. of the fuel pellet (see Fig. 1) was 3 mils. The thickness of the bottom of the tungsten cup was 75 mils and the length of the fueled region was about 1.8 inches.

The tungsten cup was prepared by chemical vapor deposition techniques. For the fuel pin of capsule V-2E, all 63 mils of the cladding consisted of tungsten prepared by the hydrogen reduction of  $WF_6$ , i. e. fluoride tungsten. The tungsten was deposited on molybdenum mandrel at  $853^{\circ}K$  and a hydrogen flow rate of 2000 cc./min. and a  $WF_6$  flow rate of 350 cc./min., the total gas pressure in the deposition chamber being 120 torr. About 5 mils of the deposit next to the surface of the molybdenum mandrel was honed off to remove the small random grains which could induce grain growth in the deposit at design operating condition. For the fuel pin of Capsule V-2F, the



TABLE 3

DIMENSIONS (IN INCH) OF FUEL PELLETS FOR  
V-2E AND V-2F CAPSULES

Fuel Sample Set	Fuel Pellet Number	Outside Diameter	Inside Diameter	Height
V-2E2 (for V-2E capsule)	1(Bottom)	.5021	.1875	.1998
	2	.5020	.1880	.2003
	3	.5020	.1875	.2001
	4	.5019	.1875	.2002
	5	.5020	.1875	.1999
	6	.5020	.1875	.2001
	7	.5020	.1880	.2000
	8	.5021	.1873	.1989
	9(Top)	.5020	.1877	.1997
V-2F1 (for V-2F capsule)	1(Bottom)	.5021	.1877	.1994
	2	.5022	.1880	.2000
	3	.5021	.1880	.2000
	4	.5020	.1880	.2001
	5	.5019	.1880	.2004
	6	.5020	.1880	.2004
	7	.5020	.1882	.1993
	8	.5021	.1880	.2000
	9(Top)	.5021	.1883	.1998

tungsten cladding consisted of a fluoride tungsten substrate of 48 mil thickness onto which 15 mils of tungsten formed by the hydrogen reduction of tungsten chloride (chloride tungsten) was deposited. The deposition of the chloride tungsten on the fluoride tungsten substrate was carried out at  $1373^{\circ}\text{K}$ , a hydrogen flow rate of 240 cc./min. and a chlorine flow rate of 230 cc./min. The tungsten chips for generating the tungsten chloride were maintained at  $1150^{\circ}\text{K}$  and the gas pressure in the deposition chamber was less than 1 torr. Chloride tungsten deposited under such conditions has been shown to possess a high degree of (110) preferred crystal orientation and a vacuum electron work function of 4.9 - 5.0 eV. Such a duplex tungsten cladding combines the advantages of a substrate of stable grain structure and high creep strength, and an emitting surface of high vacuum electron work function and superior thermionic performance.

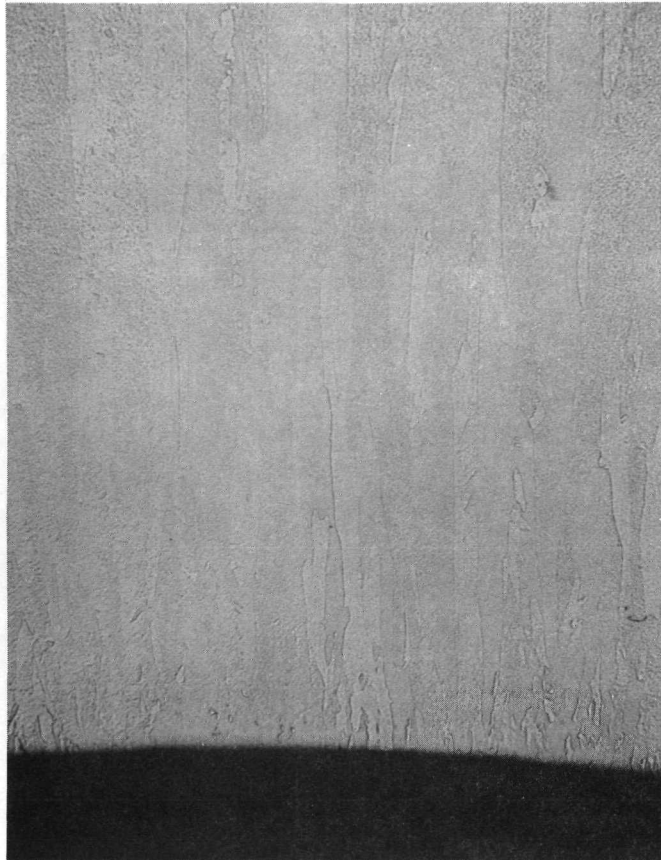
A total of four fluoride tungsten cups was deposited. Table 4 summarizes the analytical results for the impurity contents in these deposits after they were heated for 4 hours at  $2073^{\circ}\text{K}$ . No significant grain growth was observed after they were heated 4 hours at  $2073^{\circ}\text{K}$  and 4 hours at  $2473^{\circ}\text{K}$ . Sample 4434-59 was used as the fuel cup of Capsule V-2E, and Sample 4434-71 was used as the fluoride tungsten substrate of the fuel cup of Capsule V-2F. Figures 5(a) and (b) show respectively the microstructures of Sample 4434-59 after 4 hours at  $2073^{\circ}\text{K}$  and after an additional 4 hours at  $2473^{\circ}\text{K}$ . Figures 6(a) and (b) show the microstructures of Sample 4434-71 after similar heat treatments. Figure 7 contains the X-ray pole figure plots showing the

TABLE 4

IMPURITY CONTENTS IN FOUR FLUORIDE TUNGSTEN FUEL  
CUPS AFTER 4 HOURS AT 2073°K

Sample Designation	Impurity Contents (ppm)			
	F	C	N	O
4434-57	3, 4	17, 16	<1	3, 2
4434-59	10, 13	12, 13	<1	4, 3
4434-71	11, 11	9, 9	<1	3, 3
4434-73	16, 17	7, 7	<1	7, 7

Note: Duplicate analysis results are given for each sample.



M 33977-1

75X

(a) After 4 hours at  $2073^{\circ}\text{K}$

Fig. 5. Microstructures of V-2E fluoride tungsten fuel cup 4434-59 after heat treatment (Sheet 1 of 2)

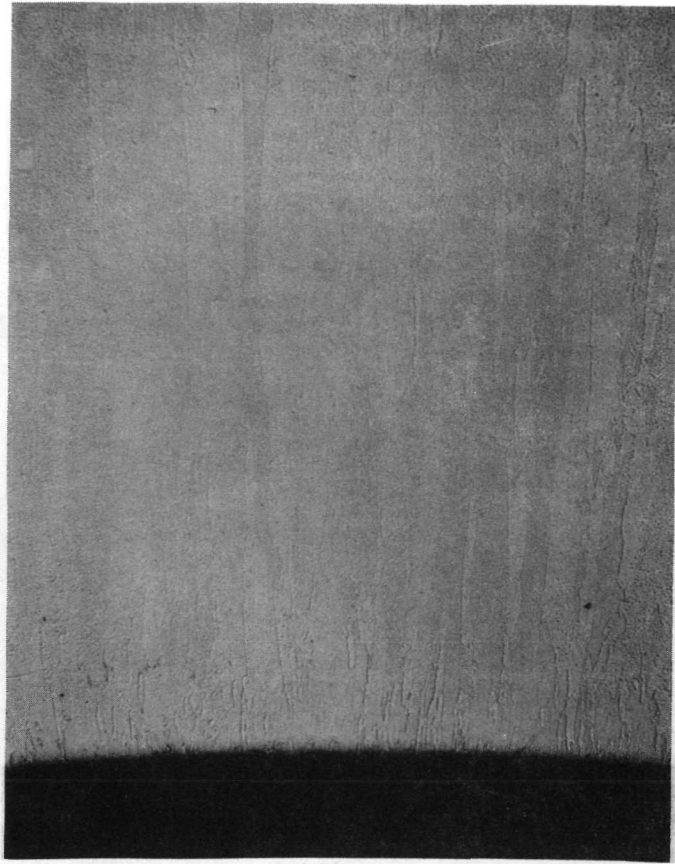


M 34010-1

75X

(b) After additional 4 hours at 2473°K

Fig. 5. Microstructures of V-2E fluoride tungsten fuel cup 4434-59 after heat treatment (Sheet 2 of 2)

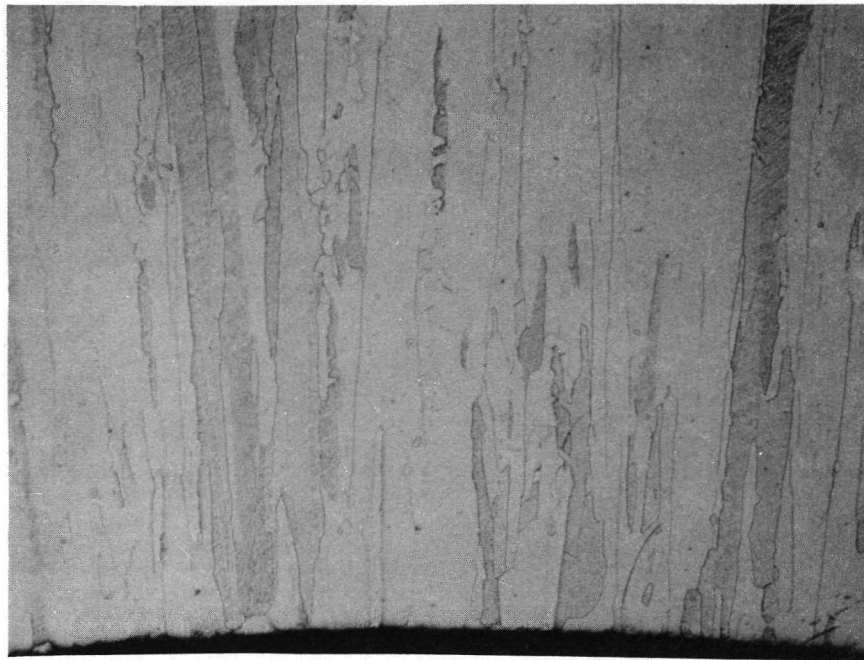


M 33997-1

75X

(a) After 4 hours at 2073°K

Fig. 6. Microstructures of fluoride tungsten substrate of V-2F duplex tungsten fuel cup 4434-71 after heat treatment (Sheet 1 of 2)



M 34065-1

75X

(b) After additional 4 hours at  $2473^{\circ}\text{K}$

Fig. 6. Microstructures of fluoride tungsten substrate of V-2F duplex tungsten fuel cup 4434-71 after heat treatment (Sheet 2 of 2)

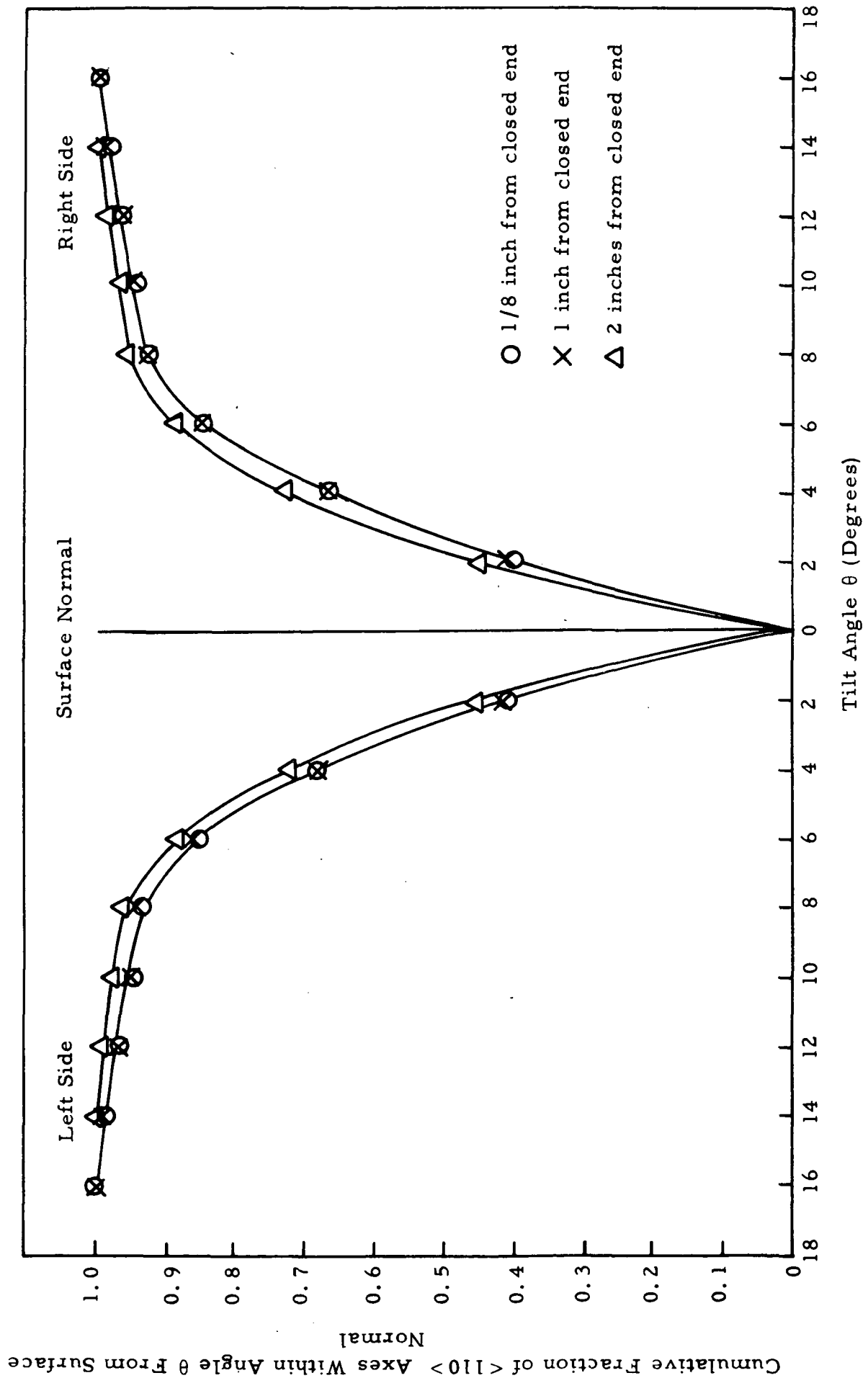


Fig. 7. Distribution of the  $\langle 110 \rangle$  axes in the chloride tungsten layer of the cladding of V-2F capsule



distributions of the  $\langle 110 \rangle$  crystal axes at three axial positions of the duplex tungsten fuel cup of Capsule V-2F after final machining and electro-polishing. The high degree of (110) preferred crystal orientation in the chloride tungsten layer of this fuel cup is evident.

Following fabrication and assembly of the fuel pins into capsules V-2E and V-2F, both capsules were delivered to the NASA Plum Brook Reactor Facility in August 1971.

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